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# Fast Pyrolysis as a Valorization Mechanism for Banana Rachis and Low-Density Polyethylene Waste

Banana rachis and low-density polyethylene (LDPE) were selected as secondary feedstocks for the study of fast pyrolysis in a free-fall reactor. The experiments were performed at 600 °C for banana rachis and 450 °C for LDPE, based on literature and thermogravimetric analysis. The gaseous products of both feedstocks present similar composition in the C1–C2 compounds, while C3 compounds are only found in LDPE. The liquid products from banana and LDPE correspond to functional groups and shorter hydrocarbons, respectively. Scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) analyses of the char showed important morphological changes to spheres in LDPE and structural changes due to thermal decomposition in the biomass. The pyrolysis char has high potential as adsorbent, encapsulation, or catalyst.

**Keywords:** Fast pyrolysis, Free-fall tube reactor, Low-density polyethylene plastics, Residual biomass, Waste management

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## 1 Introduction

Waste management is undergoing an important development toward valorization of waste streams as secondary feedstocks. Waste streams need to be considered as valuable regenerative raw materials and energy sources that require processing to be reincorporated into the production chain [1]. The valorization of these secondary feedstocks is essential for the implementation of circular economy concepts.

One technology studied in the last decade for the treatment of waste streams is the thermal decomposition in the absence of oxygen, known as pyrolysis [2]. Pyrolysis is considered a clean thermochemical conversion technology, which consists of a carbonization process where an organic material is decomposed to produce solid residual coproducts and char, non-condensable gases, and condensable liquids [3]. These products can be used as fuels, chars, chemical precursors as building blocks for the chemical industry, or directly as a renewable energy source. The yields of pyrolysis products vary depending on the primary decomposition reactions of the raw material and the secondary complex reactions of the volatiles. These yields depend on the optimization of the operational parameters such as residence time, temperature, concentration, and type of raw material. The pyrolysis occurs typically at temperatures between 400 °C and 800 °C and it has an important effect on the product distribution and properties [4].

One of the advantages of pyrolysis is the flexibility to apply a wide range of secondary feedstocks, such as residual biomass, or domestic and industrial residues like plastics, food, paper, cloth, and others. The pyrolysis of residual biomass is one the

most viable and promising techniques for the generation of renewable energy sources and chemical precursors due to its abundance and availability. The thermal decomposition of the organic components in biomass is carried out at a temperature range between 350 °C to 800 °C [5]. The long chains of hydrocarbons in the residual biomass are decomposed to smaller molecules by two pathways [6]: the first pathway involves the dehydration and charring reactions producing charcoal, CO<sub>2</sub>, and water, while the second leads to the formation of combustible volatiles by depolymerization and volatilization [7, 8].

Research of biomass pyrolysis has demonstrated that it is a profitable technique to convert rapidly solid biomass and residues which are difficult to manage, into liquid, solid, and gaseous products. García-Pérez et al. investigated woody biomass pyrolysis determining product yields and the components of biochar as function of temperature (350–600 °C) [9]. Due to

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the availability and large amounts of banana wastes generated in every production cycle, some research has been conducted to take advantage of these residues as an efficient feedstock to obtain pyrolysis products. Abdullah et al. used pyrolysis process to convert agricultural residue such as banana waste into biochar, bio-oil, and gases [10]. Taib et al. investigated the fast pyrolysis of banana pseudo-stem determining the optimum pyrolysis temperature and residence time in the range of 470–540 °C. It was found that a residence time below 1 s lead to an increase of approximately 46 % in char yield [11].

Another highly efficient and promising treatment method is the pyrolysis of plastic waste, like the highly abundant low-density polyethylene (LDPE) residues. Based on European statistics, the demand of LDPE is 17.5 %, however, its recovery rate is just 5 % [12, 13]. One of the main advantages of the decomposition of plastics in the absence of oxygen is that all final products can be used as fuels and the volume of gaseous products is low [14]. During the pyrolysis of LDPE, simpler molecules are generated due to the breakage of the long chains of polymers that suffer thermal degradation. These chemical changes occur at temperatures between 300 °C and 600 °C [15–17].

Some studies demonstrated the potential application of LDPE pyrolysis products as alternative energies and materials. Sogancioglu et al. investigated LDPE pyrolysis in a vertical chromium reactor and the samples were pyrolyzed between 300 °C and 700 °C. The oil, gas, and char obtained from pyrolysis were characterized and the application of char as additive in production of epoxy composite was evaluated [17]. Jamrloedluk and Lertsatitthanakorn pyrolyzed plastic waste at a temperature of 400–450 °C focusing on the morphology and pore properties of pyrolysis char as solid fuel [18]. Yang et al. performed copyrolysis between LDPE and three types of biomass: cedar wood, sunflower stalk, and *Falopia japonica* stem. These experiments were carried out to compare the relative yield and quality of pyrolysis products. It was demonstrated that the char yield from copyrolysis is higher than that obtained from LDPE pyrolysis. Furthermore, copyrolysis improves the hydrocarbon content of the pyrolysis product [16].

The pyrolysis behavior of two different secondary feedstocks, i.e., banana rachis and LDPE plastic, is examined to compare the thermal degradation range, product distribution, and changes in the char's morphology and chemical composition of the products. The main goal is to improve the state-of-the-art of the valorization method by fast pyrolysis of plastic wastes and residual biomass and consequently improve the implementation of circularity into the production chain.

## 2 Experimental

### 2.1 Biomass and LDPE Preparation

Pyrolysis experiments were carried out with banana rachis and LDPE plastic as raw materials. The biomass was selected based on the abundance and importance of the residual biomass produced in Ecuador [19]. Initially, the banana rachis was cut, dried, reduced to particles in a hammer mill, and then sieved to get a homogeneous particle size of less than 250  $\mu\text{m}$ .

LDPE pellets were obtained from a local plastic industry at Quito, Ecuador. This feedstock was frozen for two days at  $-80\text{ }^{\circ}\text{C}$  to increase the pellet hardness and facilitate the grinding process. The sample was sieved, classified by particle size, and consequently the range between 100 and 250  $\mu\text{m}$  was chosen to carry on the experiments. The amounts of sample used for the pyrolysis experiments were 150 mg of banana rachis and 100 mg of LDPE.

### 2.2 Pyrolysis Equipment

For this study, a free-fall fast pyrolysis setup has been designed. The equipment consists of a ceramic tubular reactor and a heating jacket with an external temperature controller model Eurotherm 3508 as indicated in Fig. 1. The ceramic reactor has an inner diameter of 25 mm and total length of 750 mm. The vertical tubular furnace is a Carbolite Gero, model VST 12/300, that reaches temperatures of 1200 °C. The samples are placed in the feeding system and carried into the reactor with an inert carrier gas stream. The carrier gas used was helium with a flow rate of 2  $\text{L min}^{-1}$ . The isothermal zone of the reactor is 30 cm and the residence time of the samples in the isothermal reaction zone is below 1 s [20]. The product streams leaving the reactor enter the separation system, which consists of a cyclone that removes the solid char and a trap where the stream of hot vapor condenses at  $-10\text{ }^{\circ}\text{C}$  with a mixture of ethylene glycol and water.

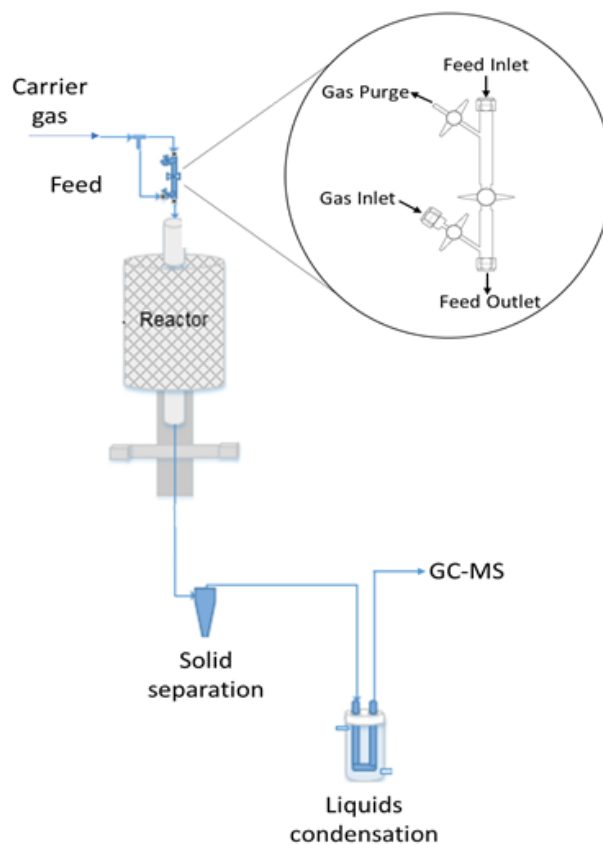


Figure 1. Diagram of the pyrolysis experimental system.

The pipeline used for the feeding system and the entrance to the separation system is of stainless steel 1/4". Finally, the condensable products are trapped and extracted with the solvent tetrahydrofuran (Loba Chemie), while the gas is injected into a gas chromatography-flame ionization detector (GC-FID) system in the case of biomass and a gas chromatography-mass spectrometry (GC-MS) system for the samples of LDPE for their characterization and quantification. The pipeline through which the gaseous products pass directly to the GC-FID is 1/8' prime] stainless-steel tubing. The liquid and solid samples are weighed and analyzed by gas chromatography, Fourier transform infrared (FTIR) spectrophotometer, a scanning electron microscopy (SEM), and thermogravimetric analysis (TGA), respectively, according to the analytical methods described next.

### 2.3 Analytical Methods

The thermal behavior of banana rachis and LDPE was investigated using TGA (Perkin Elmer). The analysis was performed to determine the temperature range of decomposition of each sample.

The solid samples' surface and morphology before and after pyrolysis process were characterized with a JSM-IT300 InTouchScope scanning electron microscope from JEOL. Also, FTIR spectroscopy analysis was performed to study the composition of the biochar and the changes in chemical structure of both samples in an infrared spectrometer Cary 630 with an attenuated total reflectance (ATR) detector.

The gaseous products obtained from the banana rachis and LDPE pyrolysis were analyzed by a Thermo Fisher Scientific gas chromatograph coupled with a flame ionization detector. Finally, the qualitative analysis of bio-oil was performed on a Thermo Fisher Scientific gas chromatograph for LDPE sample and a GC-MS Shimadzu 2010 for banana rachis both coupled with a mass spectrometer. Helium was used as carrier gas with a flow rate of 1.2 mL min<sup>-1</sup> for both samples.

In the case of banana rachis, the thermogravimetric analysis was carried out under a nitrogen atmosphere with a flow rate of 2 mL min<sup>-1</sup>. The sample weight was 50 mg and it was heated from 25 °C until 600 °C at a rate of 10 °C min<sup>-1</sup>. The surface and morphology changes were observed in by SEM with an amplification of 100× and 500×. The infrared (IR) spectra were recorded in the spectral range between 4000 and 500 cm<sup>-1</sup>. The operational conditions for the gaseous injections in the FID detector of the gas chromatographer were as follows: sampling valve time 3–10 min, inlet temperature 80 °C, detector temperature 200 °C, and the oven at was held at 30 °C for 5 min and heated until 200 °C at a rate of 10 °C min<sup>-1</sup>.

The qualitative analysis of liquid products was realized in a stainless-steel column MXT-1 and the conditions were: MS detector temperature 200 °C, oven heated from 30 °C and held 5 min until it reached 300 °C with a rate of 3 °C min<sup>-1</sup>. The split flow was 30 mL min<sup>-1</sup> with a split rate of 15. The chromatograms were obtained in full-scan mode and peaks were analyzed using the Nyst library.

LDPE samples analyzed in the thermogravimetric analyzer used a nitrogen atmosphere with a flow of 20 mL min<sup>-1</sup> and

were heated from 25 °C to 700 °C with a rate of 10 °C min<sup>-1</sup>. The LDPE char's morphology was studied and compared at different temperatures with amplifications of 50× and 100×.

FTIR spectroscopy was carried out in a wavenumber range between 4000 and 650 cm<sup>-1</sup>. Liquid oil was characterized by a gas chromatograph using a TG-5MS column with nonpolar phase of 5 % diphenyl and 95 % dimethyl polysiloxane. The conditions set for the analyses of LDPE were: oven temperature programmed at 50 °C for 1 min, then heated to 280 °C at a rate of 4 °C min<sup>-1</sup>, holding this temperature for 10 min; injector temperature 320 °C with a splitless mode set up; ion source and MS transfer line temperature 230 °C and 320 °C, respectively.

The volatile compounds of gaseous products were identified using a TG Bond Q column. The column phase was nonpolar with 100 % divinyl benzene and the column flow was 2.1 mL min<sup>-1</sup>. The GC oven temperature was held at 30 °C for 5 min and then heated until it reached 200 °C with a rate of 10 °C min<sup>-1</sup>. Detector and injector temperatures were 200 °C and 250 °C, respectively. Additionally, the injector was operating at split mode with a split flow of 21 mL min<sup>-1</sup>.

## 3 Results and Discussion

### 3.1 Product Distribution of the Pyrolysis of Banana Rachis and LDPE

The pyrolysis experiments of residual biomass and LDPE plastic were carried out at 600 °C and 450 °C, respectively. These temperatures were chosen based on literature studies and following the thermal degradation behavior of LDPE and banana rachis as presented in Fig. 2. The thermograph of LDPE in Fig. 2a showed that the thermal decomposition ranged between 400 °C and 500 °C similar to the studies conducted by Uddin et al. [21] pyrolyzing LDPE in a batch reactor at 430 °C and Aguado et al. [22] who used the same type of reactor to pyrolyze LDPE at 450 °C.

Regarding the banana rachis thermograph in Fig. 1b, it can be stated that after an initial humidity loss around 100 °C the samples thermally decompose steadily in the range between 200 °C and 500 °C. This can be corroborated with a previous study by Montoya et al. [23] that involved pyrolysis of residual biomass at temperatures between 400 °C and 600 °C or the study by Chatterjee et al. [24] that determined 500–800 °C as the adequate pyrolysis temperature range to produce biochar products. According to Guizani [25], a pyrolysis temperature below 600 °C favors the production of bio-oil and biochar products, while temperatures above 600 °C maximize the production of pyrolysis gas.

Additionally, there are studies that confirm biochar production having three stages, i.e., pre-pyrolysis, main pyrolysis, and formation of carbonaceous soil products. The last stage, which occurs at temperatures above 500 °C, decomposed lignin and other organic matter. It is known that higher lignin content results in a higher biochar yield because it produces more charcoal and tar [26, 27].

Based on the literature and considering that the aim of this study is to carry out an analysis of the pyrolysis products with a special focus on maximization and morphology of the solid

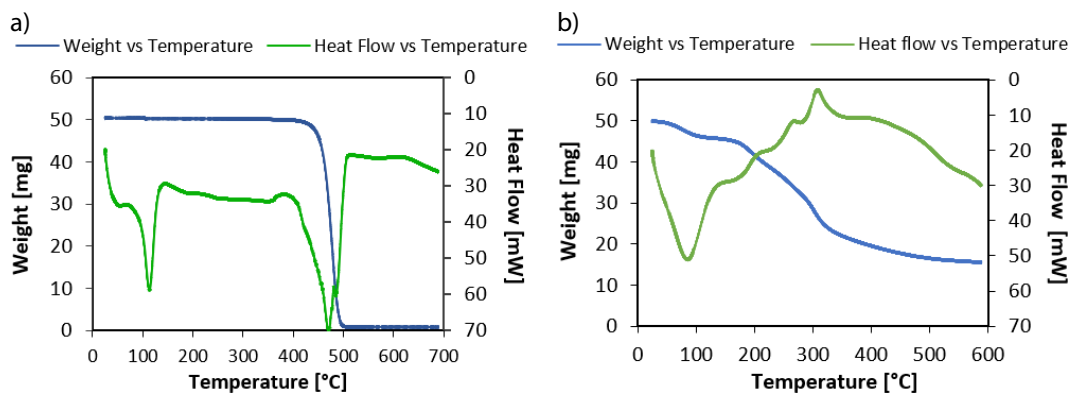


Figure 2. TGA analysis of (a) LDPE and (b) raw biomass samples.

products, the product distribution of LDPE and banana rachis pyrolysis were studied at 450 °C and 600 °C, respectively.

The distributions of the liquid, gaseous, and solid products were determined from the different samples after pyrolysis. The product distributions obtained from pyrolysis of biomass and LDPE at 600 °C and 450 °C, respectively, are indicated in Fig. 3. In the case of LDPE, a char yield of 43% was found, while banana rachis pyrolysis revealed a biochar yield of 71%. The yields achieved for banana rachis pyrolysis for gases and oil were 7% and 22%, respectively, while LDPE presented gas and oil yields of 55% and 2%.

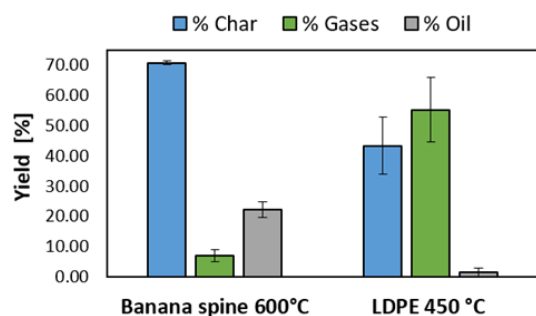


Figure 3. Pyrolysis product distribution of banana rachis and LDPE.

These results are in accordance with pyrolysis analyses reported in literature. A study performed in Nigeria which used plantain stem revealed a char yield of 83% and determined that the results were consistent with the finding of Bridgewater et al. that biomass with a high content of lignin will give more biochar than bio-oil [28,29]. In general, banana stem characterization presented values of lignin between 15% and ~35% [30–32]. Fernandes et al. [17] pyrolyzed pseudo-stem banana samples at 500 °C and obtained a char yield of 58% while Abdullah et al. reported a value of ~52% [33, 34].

In the case of LDPE, the pyrolysis compounds are involved in a free-radical polymerization reaction that causes the formation of char. Char production is directly influenced by the temperature. Tiikma et al. examined this effect on the thermal processing of LDPE [35]. They found that the optimum oil yield was obtained at 450 °C and there was also a significant formation of char at this temperature. Additionally, some studies per-

formed at a temperature range between 450 °C and 500 °C reported a char yield of 45.3% and 52.9% [17, 36].

The obtained product distribution can be compared to reports found in literature. The proposed pyrolysis mechanisms for biomass and LDPE feedstocks can be used to understand the results in this study. Regarding the reaction kinetic models for the thermal degradation of LDPE, Kple et al. [37] presented the radical chain mechanism as the accepted mechanism to support the thermal degradation of polyethylene. In addition, a simple step reaction model was described where two products are obtained from the thermal degradation of LDPE: char and volatiles [37].

Kayacan and Dogan reported similar results, considering a first-order reaction model that provided information on activation energy and pre-exponential factor [38]. A mechanistic model for biomass pyrolysis suggests that fast pyrolysis consists of primary activation, fragmentation reactions, and secondary vapor-phase cracking reactions [39,40]. Calonaci et al. analyzed and proposed a kinetic model for the fast pyrolysis of biomass and compared these predictions to experimental measurements [41]. Ojha et al. evaluated the apparent kinetics of fast pyrolysis of lignocellulosic biomass like rice straw, pine wood, and fruit bunch. The study determined the kinetic parameters using empirical models [42].

Other studies focused on individual biomass components like cellulose, hemicellulose, and lignin in order to make the understanding of biomass pyrolysis kinetics easier. The simplified kinetics of these components were combined with computational fluid dynamics to obtain product distributions for process optimizations in different reactor configurations [43, 44].

### 3.2 Composition of Liquid and Gaseous Products of Biomass and LDPE

The liquid products were obtained as explained in the analytical methods. The principal compounds identified in bio-oil samples of banana rachis were aromatic rings, emi-acetyl, aldehydes with an aromatic ring, esters, ether, and cyclic acetyl. These are compounds with high added value. Literature about bio-oil composition determined that it is a complex organic mixture which contains highly oxygenated compounds. These compounds include water, alcohols, carboxylic acids like acetic

acid or formic acid, aldehydes, ketones, pyrones, furans, phenols, furan carboxaldehydes, sugars, alkenes, aromatics, nitrogen compounds, miscellaneous oxygenates, and higher-molecular-weight oligomers [45].

For the liquid oil of LDPE sample, no individual peaks were identified. A carbon range between C14 to C35 was observed. These results were supported by literature which establishes that the typical carbon range for oil is C20–C35. The range of diesel fuel is observed between C10 and C28. This information confirms that liquid oil from LDPE has a higher potential as energy source because it has a large number of carbon chains and a high content of aromatic hydrocarbons [46].

Regarding the gaseous qualitative analyses, methane, ethane, and acetylene were obtained from the pyrolysis of both LDPE and banana rachis. On the other hand, propane and propylene were observed only from the LDPE oil samples, while ethylene was only found in the banana rachis gaseous product. The results found by analysis of gaseous products from LDPE pyrolysis are supported by other authors, i.e., Encinar and Gonzalez determined that the gas composition from pyrolysis of polyethylene contained alkanes and alkenes up to C3 and hydrogen [47].

Additionally, a study of co-pyrolysis of biomass and plastic waste identified in the gas phase alkanes with a maximum of four carbons and two additional compounds, i.e., CO and CO<sub>2</sub> that were obtained as result of the pyrolysis of biomass [48]. In the case of the pyrolysis of residual biomass, studies revealed that the most relevant components of pyrolytic volatiles are H<sub>2</sub>O, CO<sub>2</sub>, CO, H<sub>2</sub>, and CH<sub>4</sub> [49, 50].

### 3.3 Solid Product Morphology of Biomass and LDPE

Char samples before and after pyrolysis of LDPE and banana rachis were analyzed using a scanning electron microscope and an FTIR spectrophotometer. FTIR is applied to analyze the change in the chemical composition of the samples before and after the pyrolysis process, while SEM is employed to observe the morphology and particle size distribution. The non-pyrolyzed sample and char at 600 °C for banana rachis and 450 °C for LDPE were analyzed to identify and compare bonds. The FTIR spectra from both samples are illustrated in Fig. 4.

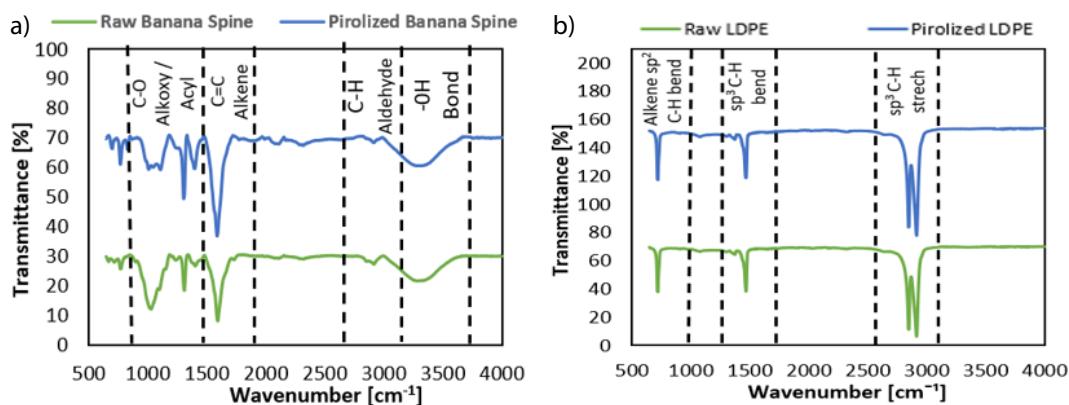


Figure 4. FTIR spectra of (a) banana rachis and (b) LDPE samples before and after pyrolysis.

From the spectra presented in Fig. 4a it can be observed that there are significant changes in the C–O alkoxy (1027.90 cm<sup>-1</sup>), C–O acyl (1236.17 cm<sup>-1</sup>), C=C alkene (1622.95 cm<sup>-1</sup>), C–H aldehyde (2917.19 cm<sup>-1</sup>), and –OH bonds (3318.80 cm<sup>-1</sup>) after pyrolysis. Double-carbon and C–O acyl bonds increase after pyrolysis, while the C=O carbonyl and hydroxyl groups and C–O alkoxy bonds decrease. In contrast, the spectra in Fig. 4b reveals a peak in the range of 2916 and 2849 cm<sup>-1</sup> that represents alkanes with C–H stretching vibration, and the peaks located between 1645 and 1464 cm<sup>-1</sup> indicates aromatic groups with C=C stretching vibration. Spectra of raw and pyrolyzed LDPE are similar and there is not significant change between the peak's areas.

The raw biomass and LDPE as well as the pyrolyzed samples were analyzed by SEM to evaluate morphological changes. The SEM images are presented in Fig. 5. Samples of raw banana rachis in Figs. 5a and 5b indicate a fibrous structure before carbonization. After pyrolysis, the banana rachis sample particles agglomerate due to coke formation and their structure becomes more porous. Therefore, the structure of the residual biomass after pyrolysis exhibits significant changes in the morphology since the fibers in the external layer partially

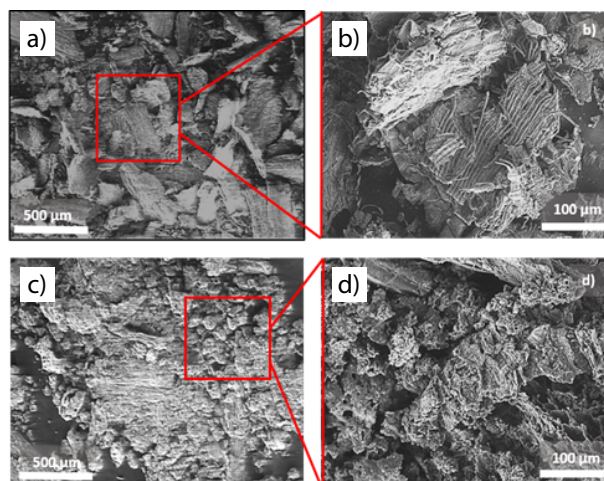
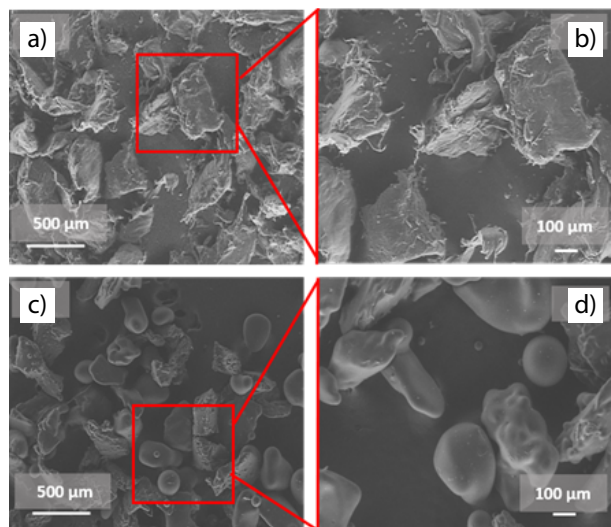


Figure 5. Morphology analysis with SEM of banana rachis (a), (b) before pyrolysis; (c), (d) after pyrolysis with a magnification of 50x and 250x.

disappear. Also, particles in the raw sample are divided, porosity increases, and the char particles present a granular appearance.

According to literature, the temperature of pyrolysis had a notable impact on the structure and the morphology of biomass char. It has been found that high pyrolysis temperatures cause changes in biochar surface area and porosity [51]. This happens due to the decomposition of organic matter and the formation of micropores [52].

The SEM images of raw and pyrolyzed LDPE are presented in Fig. 6. Raw LDPE and char obtained at 450 °C were considered for this analysis. Due to the high tensile strength to which the raw LDPE was subjected during the milling process, the particles presented small irregular filaments as illustrated in Figs. 6a and 6b. After pyrolysis at 450 °C, most filaments reshaped into spheres as it can be seen in Figs. 6c and 6d, with few exceptions of irregular particles that are assumed to be coke. The solids collected from the walls look more like melted wax-like plastics, indicating that the solid did not pyrolyze completely and only the molecules at the outer particle surfaces cracked. SEM results of LDPE demonstrate that LDPE acquires a spheric shape after being exposed to high temperatures. The SEM images show a significant morphology difference between irregular raw plastic and pyrolyzed spheres.



**Figure 6.** Morphology analysis with SEM of LDPE (a), (b) before pyrolysis; (c), (d) after pyrolysis with a magnification of 50× and 100×.

## 4 Conclusions

The secondary feedstocks banana rachis and LDPE were pyrolyzed in a tubular free-fall reactor with a heating jacket at different temperatures selected based on TGA. The principal products obtained were char, oil, and non-condensable volatiles. The product distribution of biomass showed a char yield of 71 % approximately at 600 °C while LDPE samples obtained a char yield of 42 % at 450 °C.

Gaseous and liquid products were analyzed in a GC-FID/MS chromatograph. The biomass gases are methane, ethane, ethylene, and acetylene. The liquid products include the following types of organic compounds: aromatic rings, emi-acetyl, aldehyde with an aromatic ring, esters, ether, and cyclic acetyl. The solid products show a change in the fibrous external layer into granular particles with an increase of the porosity. The LDPE plastics composition of the gas phase is methane, ethane, acetylene, propane, and propylene. The liquid oils present a carbon range from C14 to C35.

The solids collected from the cyclone are perfect hollow spheres. The solids collected from the walls look more like melted wax-like plastics, indicating that the solid did not pyrolyze completely and only the molecules at the outer particle surfaces cracked. Finally, the FTIR spectra of biomass reveals a chemical change in the structure of the C–O alkoxy bonds, hydroxyl groups (–OH), C–O acyl, C=C alkene, and C–H aldehyde. LDPE spectra present a small variation of the peaks proportion after pyrolysis at 600 °C.

It can be concluded that the pyrolysis of LDPE and residual biomass exhibits a significant difference in product distribution, composition, and morphology of the solids. For this reason, these feedstocks will be further investigated with a more rigorous analysis where the liquid and gaseous products can be quantified. Moreover, it is important to compare the pyrolysis of banana rachis and LDPE residual plastics at different temperatures. This comparison will be done to continue with the investigation of copyrolysis of both feedstocks since it has been demonstrated that there is a positive effect when these mixtures are pyrolyzed. Due to the complexity of the biomass and LDPE structure as well as the pyrolysis product molecules, more experiments must be carried out to obtain the apparent kinetic parameters. These parameters are useful for the optimization of the thermal behavior, reactor design, and operating conditions of the pyrolysis process.

From this preliminary research, it can be stated that plastics produce chemical precursors useful in the petrochemical industry, while biomass generates a vast variety of high-added-value products as green chemicals, solvents, aromatics, acids, alcohols, and other compounds. The solid products also have interesting applications as adsorbents, catalysts, additives, among others. Finally, copyrolysis could be a green solution to reduce the poor disposal of plastic and organic wastes of the industries like banana producers. The large amounts of discarded plants and the LDPE bags used to cover the banana fruits can be recycled as raw materials for thermochemical processes.

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## Abbreviations

|        |                                                   |
|--------|---------------------------------------------------|
| ATR    | attenuated total reflectance                      |
| FTIR   | Fourier transform infrared spectroscopy           |
| GC-FID | gas chromatography with flame ionization detector |
| GC-MS  | gas chromatography with mass spectrometry         |
| IR     | infrared                                          |
| LDPE   | low-density polyethylene                          |
| SEM    | scanning electron microscopy                      |
| TGA    | thermogravimetric analysis                        |

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